



(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention
of the grant of the patent:
31.10.2001 Bulletin 2001/44

(21) Application number: **96928911.5**

(22) Date of filing: **16.08.1996**

(51) Int Cl.7: **H01B 3/44**

(86) International application number:
PCT/US96/13356

(87) International publication number:
WO 97/07515 (27.02.1997 Gazette 1997/10)

(54) **FLUOROPOLYMER EXTRUSION PROCESS**
EXTRUSIONSVERFAHREN VON FLUORKUNSTSTOFFEN
PROCEDE D'EXTRUSION DE FLUOROPOLYMERES

(84) Designated Contracting States:
DE FR GB IT NL

(30) Priority: **17.08.1995 US 2404 P**
23.02.1996 US 12130 P
23.07.1996 US 685083

(43) Date of publication of application:
03.06.1998 Bulletin 1998/23

(73) Proprietor: **E.I. DU PONT DE NEMOURS AND**
COMPANY
Wilmington Delaware 19898 (US)

(72) Inventor: **BLAIR, Leslie, Mitchell**
Parkersburg, WV 26101-9240 (US)

(74) Representative: **Matthews, Derek Peter et al**
Frank B. Dehn & Co.,
European Patent Attorneys,
179 Queen Victoria Street
London EC4V 4EL (GB)

(56) References cited:
EP-A- 0 318 876 **EP-A- 0 320 901**

Note: Within nine months from the publication of the mention of the grant of the European patent, any person may give notice to the European Patent Office of opposition to the European patent granted. Notice of opposition shall be filed in a written reasoned statement. It shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

Description**RELATED APPLICATIONS**

- 5 [0001] This application is a Continuation of Provisional Application Serial No. 60/012,130 filed February 23, 1996 and a Continuation-In-Part of Provisional Application No. 60/002,404 filed August 17, 1995.

FIELD OF THE INVENTION

- 10 [0002] This invention is in the field of processes for fabricating copolymers of tetrafluoroethylene in the molten state.

BACKGROUND OF THE INVENTION

- 15 [0003] Carlson in U.S. Patent 4,029,868 (1977) discloses the improvement of melt-fabricable copolymers of tetrafluoroethylene (TFE) and hexafluoropropylene (HFP) containing 4-12 wt% HFP by incorporation of 0.5-3 wt% of either perfluoro(ethyl vinyl ether) or perfluoro(propyl vinyl ether) into the copolymer. The resultant terpolymer is also melt-fabricable, has improved high temperature tensile strength without diminished flex life, and exhibits snap-back so as to be useful as heat shrinkable tubing. The polymerization is carried out using the solvent process or the aqueous dispersion process using added solvent as described by Carlson in U.S. Patents 3,528,954 and 3,642,742, respectively.
- 20 The HFP content corresponds to an infrared HFPI index (HFPI) range of 0.9 to 2.7, using the multiplicative factor 4.5 disclosed to convert HFPI to HFP content in wt%. Example 13 discloses a TFE/HFP/PEVE terpolymer (4.5 wt% HFP and 1.2 wt% PEVE) as providing high toughness, but most of the Examples are directed to TFE/HFP/PPVE terpolymer.
- [0004] McDermott & Pierkarski in SIR H310 (1986), subsequent to Carlson, demonstrated the preference for TFE/HFP/PPVE terpolymer by disclosing only this polymer for achieving improved stress crack resistance, obtaining this
- 25 improvement with an HFP content of 9-17 wt% and PPVE content of 0.2-2 wt%. The HFP content corresponds to an HFPI range of about 2.8-5.3, using the multiplicative factor 3.2 disclosed to convert HFPI to HFP content in wt%. The non-aqueous polymerization procedure of the Carlson '954 and '868 patents are referenced. Aqueous dispersion polymerization is also disclosed, with optional addition of unreactive fluorocarbon phase to promote monomer diffusion or to solubilize the initiator. The goal of SIR H310 was to increase stress crack resistance at the same copolymer melt
- 30 viscosity or to allow a faster extrusion rate while keeping the stress crack resistance at a high level. The problem solved by SIR H310 involved a trade off between extrusion rate and stress crack resistance. Provision of copolymer having a melt viscosity which enabled faster extrusion was done at some sacrifice in stress crack resistance.
- [0005] As is common in the field, both Carlson and McDermott & Piekarski base HFP content on measurement of HFPI. This quantity was introduced by Bro & Sandt in U.S. Patent 2,946,763 which pertains to TFE/HFP copolymers.
- 35 Bro & Sandt also introduced the multiplicative factor 4.5 to obtain HFP content in wt% from HFPI. While recent calibrations have led to different multiplicative factors, HFPI values deduced from infrared measurements at different times are generally regarded as reliable.
- [0006] A TFE/HFP copolymer resin that can be extruded faster without sacrifice of stress crack resistance is desired.

40 **SUMMARY OF THE INVENTION**

- [0007] Copolymers of tetrafluoroethylene, hexafluoropropylene, and perfluoro(ethyl vinyl ether) permit melt processing rates that are surprisingly greater than similar copolymer containing perfluoro(propyl vinyl ether). Thus, the present invention provides a process of extruding and melt drawing a tetrafluoroethylene copolymer resin to form insulation on
- 45 an electrical conductor, wherein said copolymer is a partially-crystalline copolymer comprising tetrafluoroethylene, hexafluoropropylene in an amount corresponding to HFPI of from 2.0 to about 5.3, and from 0.2% to 3% by weight of perfluoro(ethyl vinyl ether).

DETAILED DESCRIPTION OF THE INVENTION

- 50 [0008] It has been discovered that TFE/HFP/PEVE copolymer resin can be extruded at rates surprisingly higher than possible with corresponding TFE/HFP/PPVE copolymer. In the application of TFE/HFP/PEVE resin as wire insulation on metal conductor by a process involving melt extrusion and melt draw, it is possible to run at wire speeds about 1.5x as fast as with counterpart resin containing PPVE, a very substantial and commercially significant improvement.
- 55 [0009] As illustrated by examples to follow, TFE/HFP/PEVE copolymer used in the process of this invention remarkably exhibits no melt fracture in capillary rheometry at shear rates substantially in excess of the shear rate at which counterpart resin containing PPVE instead of PEVE exhibits gross melt fracture.
- [0010] As also illustrated by examples to follow, TFE/HFP/PEVE copolymer of this invention can also be subjected

to greater, and more rapid, melt draw than counterpart resin containing PPVE. Melt draw ("drawing down") is a technique employed in processing of certain fluoropolymers to enhance rate otherwise limited by melt fracture in extrusion, i.e., by using a die with a relatively large opening and drawing the extruded melt to desired final dimensions. Melt draw is commonly characterized by the draw down ratio calculated as the ratio of the cross-sectional area of the die opening to the cross-sectional area of the finished extrudate.

[0011] The extruding and melt drawing process of this invention can be carried out using equipment and procedures generally used for previously known melt-fabricable TFE copolymers. Such equipment and procedures are known in the art, and are summarized, for example, in "Extrusion Guide for Melt-Processible Fluoropolymers" (3/93, DuPont). The process of this invention uses TFE/HFP/PEVE copolymer resin.

[0012] The TFE/HFP/PEVE copolymers used in the process of this invention have HFP content preferably corresponding to HFPI = 2.2-5.3, more preferably HFPI = 2.8-4.7. For reasons of productivity in polymerization, HFP content corresponding to HFPI = 2.8-4.1 is especially preferred. HFPI is determined by an infrared method outlined below.

[0013] PEVE content of the copolymers of this invention is in the range 0.2-3 wt%, preferably 0.4-2 wt%. Another preferred PEVE content in the copolymer is 0.5-2.5 wt%. PEVE content in the copolymer is determined by an infrared method, also outlined below.

[0014] One skilled in the art will recognize that one or more additional copolymerizable monomers can be incorporated in the TFE/HFP/PEVE copolymers of this invention. The amount of such additional monomer will be such that the resultant copolymer remains partially crystalline, as indicated by detection of a melting endotherm by differential scanning calorimetry for resin as-polymerized, i.e., for resin that has not been previously melted.

[0015] Copolymers of this invention generally have melt viscosity (MV) in the range $0.5-50 \times 10^3$ Pa·s. MV in the range $1-10 \times 10^3$ Pa·s is preferred.

[0016] The TFE/HFP/PEVE copolymers of this invention can be made by any method of polymerization that yields generally homogeneous copolymer composition. Such methods include polymerization in aqueous media, polymerization in non-aqueous media, and polymerization in mixed media. Organic liquids used in the latter two polymerization systems commonly are halogenated compounds. In light of current environmental concerns about such compounds, aqueous dispersion polymerization is preferred. Such a process is disclosed, for example, for TFE/HFP/PPVE copolymer in SIR H130.

[0017] For aqueous polymerization, a broad range of temperatures can be used. Because of the low reactivity of HFP relative to that of TFE, higher temperatures are advantageous, such as temperatures in the range of about 95°-115°C. Temperature in the range 98°-108°C is preferred for making the copolymers of this invention by the aqueous semibatch process used in the examples below. Surfactants used in emulsion polymerization appear to be less effective at temperatures above 103°-108°C and there is a tendency to lose dispersion stability.

[0018] Surfactants suitable for use in dispersion polymerization of TFE/HFP copolymers can be used. Such surfactants include, for example, ammonium perfluorooctanoate (C-8), ammonium perfluorononanoate (C-9), and the perfluoroalkyl ethane sulfonic acids and salts thereof disclosed in U.S. Patent 4,380,618.

[0019] Initiators commonly employed in emulsion polymerization of TFE copolymers are water-soluble free-radical initiators such as ammonium persulfate (APS), potassium persulfate (KPS), or disuccinic acid peroxide. APS and/or KPS is preferred.

[0020] After the reactor is charged with water, surfactant and monomers, heated to the chosen temperature, and agitation is started, a solution of initiator is added at a prescribed rate to initiate polymerization. A pressure drop is the usual indicator that polymerization has started. Then, TFE addition is started and controlled according to the scheme chosen to regulate the polymerization. An initiator solution, which can be the same as or different from the first initiator solution, is usually added throughout the reaction.

[0021] There are several alternatives for regulating the rate of TFE/HFP copolymerization, and these are applicable for polymerizing the TFE/HFP/PEVE copolymers of this invention. It is common with most alternatives first to precharge all HFP monomer and then to add TFE to the desired total pressure. Additional TFE is then added after initiator injection and reaction kickoff to maintain the chosen pressure. The TFE may be added at a constant rate, with agitator speed changed as necessary to increase or decrease actual polymerization rate and thus to maintain constant total pressure. Alternatively, the total pressure and the agitator speed may both be held constant, with TFE added as necessary to maintain the constant pressure. A third alternative is to carry out the polymerization in stages with variable agitator speed, but with steadily increasing TFE feed rates.

[0022] The HFP monomer is much less reactive than the TFE monomer so that the HFP/TFE ratio must be kept high to assure a high incorporation of HFP.

[0023] The PEVE can be incorporated into the copolymer by either pre-charge, pre-charge plus subsequent addition (pumping), or pumping of the PEVE into the reactor. The reactivity of PEVE relative to TFE is such that TFE/HFP/PEVE copolymer that is satisfactorily uniform with respect to PEVE incorporation can be obtained if PEVE is precharged to the reactor, and this is preferred.

EXAMPLES

[0024] Fluoropolymer compositions were determined on 0.095-105 mm thick films pressed at 300°C, using Fourier transform infrared spectroscopy. For HFP determination, the method described in U.S. Patent 4,380,618 was used. In applying this method, the absorbances of bands found at about 10.18 micrometers and at about 4.25 micrometers were used. HFP content is expressed as an HFP index (HFPI), the ratio of the 10.18 micrometers absorbance to the 4.25 micrometers absorbance. HFP content in wt% was calculated as $3.2 \times \text{HFPI}$.

[0025] PEVE was determined from an infrared band at 9.17 micrometers. PEVE content in wt% was calculated as $1.3 \times$ the ratio of the 9.17 micrometers absorbance to 4.25 micrometers absorbance. The absorbance at 9.17 micrometers was determined using a TFE/HFP dipolymer reference film to subtract out a strong absorbance that overlies the 9.17 micrometers band. The 4.25 micrometers internal thickness absorbance was determined without use of reference film.

[0026] Melt viscosities of the fluoropolymers were determined by ASTM method D1238-52T modified as described in U.S. Patent 4,380,618.

[0027] Thermal characteristics of fluoropolymer resins were determined by DSC by the method of ASTM D-4591-87. The melting temperature reported is the peak temperature of the endotherm on second melting.

[0028] Average size of polymer particles as polymerized, i.e., raw dispersion particle size (RDPS), was measured by photon correlation spectroscopy.

[0029] The standard MIT folding endurance tester described in ASTM D-2176 was used for determining flex life (MIT Flex Life). Measurements were made using compression-molded films that were quenched in cold water. Film thickness was $0.008 \pm .0005$ inch (0.20 ± 0.013 mm).

[0030] In the following, unless otherwise stated, stated solution concentrations are based on combined weight of solvent water and of solute(s). Stated concentrations of polymer solids in dispersions are based on combined weights of solids and aqueous medium, and were determined gravimetrically, i.e., by weighing dispersion, drying, and weighing dried solids, or by an established correlation of dispersion specific gravity with the gravimetric method.

Example 1

[0031] A cylindrical, horizontal, water-jacketed, paddle-stirred, stainless steel reactor having a length to diameter ratio of about 1.5 and a water capacity of 80 parts by weight was charged with 50 parts of demineralized water and 0.36 part of a 20 wt% solution of ammonium perfluorooctanoate surfactant (C-8, Fluorad® FC-143, 3M) in water. With the reactor paddle agitated at 35 rpm, the reactor was heated to 65°C, evacuated, purged with TFE, and evacuated again. The reactor temperature then was increased to 103°C, and 0.22 part (calculated from 711 mmHg pressure rise) of liquid PEVE was injected into the reactor. After the temperature had become steady at 103°C, HFP was added slowly to the reactor until the pressure was 437 psig (3.1 MPa). Then TFE was added to the reactor to achieve a final pressure of 600 psig (4.2 MPa). Then 0.39 part of a freshly prepared aqueous initiator solution containing 0.80 wt% of ammonium persulfate (APS) and 0.80 wt% potassium persulfate (KPS) was charged into the reactor at 0.1 part/min. Then, this same initiator solution was pumped into the reactor at 0.013 part/min for the remainder of the polymerization. After polymerization had begun as indicated by a 10 psig (0.07 MPa) drop in reactor pressure, additional TFE was added to the reactor to maintain pressure constant at 600 psig (4.2 MPa) until a total of 17.5 parts of TFE had been added to the reactor after kickoff. Total reaction time was 175 min with a TFE addition rate of 0.1 part/min. The reaction rate was maintained constant by adjusting the agitator speed. At the end of the reaction period, the TFE feed and the initiator feed were stopped, and the reactor was cooled while maintaining agitation. When the temperature of the reactor contents reached 90°C, the reactor was slowly vented. After venting to nearly atmospheric pressure, the reactor was purged with nitrogen to remove residual monomer. Upon further cooling, the dispersion was discharged from the reactor at below 70°C. Solids content of the dispersion was 28.1 wt% and raw dispersion particle size (RDPS) was 0.188 μm . After mechanical coagulation, the polymer was isolated by compressing excess water from the wet polymer and then drying this polymer in a 150°C convection air oven. The TFE/HFP/PEVE terpolymer had an MV of 2.70×10^3 Pa-s, an HFPI of 4.06 (13.1 wt% HFP), a PEVE content of 0.68 wt%, and a melting point of 241°C. This polymer was stabilized by heating at 360°C for 1.5 hr in humid air containing 13 mol% water. A film molded of stabilized copolymer resin then had an MIT Flex Life of 10,900 cycles to break, showing that PEVE terpolymers of this invention have good flex life.

Control A

[0032] The procedure of Example 1 was generally repeated except that 0.33 part of PPVE was used instead of PEVE, HFP was charged to a pressure of 435 psig (3.1 MPa), and the pumping rate for initiator solution throughout the batch was 0.009 part/min. Solids content of the dispersion was 29.9 wt% and raw dispersion particle size (RDPS) was 0.176 μm . The TFE/HFP/PPVE terpolymer had an MV of 2.08×10^3 Pa-s and a melting point of 252°C. By high-

temperature ^{19}F NMR measurement, it was determined that HFP content was 12.0 wt% (corresponding to HFPI = 3.75) and PPVE content was 0.85 wt%. A film molded of stabilized copolymer resin then had an MIT Flex Life of 6200 cycles to break.

5 **Examples 2-7**

[0033] The procedure of Example 1 was essentially followed, except for differences noted in Table 1. The notation "nc" indicates no change from Example 1. Product properties are also summarized in the Table. The data show that PEVE terpolymers of this invention have excellent flex life.

10

15

20

25

30

35

40

45

50

55

Table 1. Conditions and Results for Examples 2-7

| Example: | 2 | 3 | 4 | 5 | 6 | 7 |
|---------------------------------------|-------|-------|-------|-------|-------|-------|
| Run conditions: | | | | | | |
| PEVE precharge (part) | 0.21 | 0.31 | 0.32 | 0.32 | 0.36 | 0.41 |
| HFP pressure (MPa) | nc | nc | 2.9 | 2.5 | 2.5 | nc |
| Initiator pumping (part/min) | 0.011 | 0.009 | 0.011 | 0.014 | 0.012 | 0.006 |
| Dispersion properties: | | | | | | |
| Solids (wt%) | 30.3 | 31.2 | 28.1 | 24.2 | 24.8 | 31.2 |
| RDPS (μm) | 0.197 | 0.184 | 0.182 | 0.196 | 0.194 | 0.180 |
| Resin properties: | | | | | | |
| MV ($10^3 \text{ Pa}\cdot\text{s}$) | 3.28 | 3.32 | 2.86 | 4.09 | 3.32 | 5.60 |
| HFPI | 4.16 | 3.73 | 3.59 | 3.16 | 3.13 | 3.69 |
| HFP content (wt%) | 13.3 | 11.9 | 11.5 | 10.1 | 10.0 | 11.8 |
| PEVE content (wt%) | 0.60 | 1.40 | 1.06 | 1.00 | 1.27 | 1.40 |
| Melting point ($^{\circ}\text{C}$) | 248 | 243 | 245 | 253 | 253 | 245 |
| MIT Flex Life (cycles) | 12700 | 15400 | 8980 | 4090 | 5150 | 34100 |

Example 8

[0034] TFE/HFP/PEVE terpolymer resin produced by the general procedure of Example 1 and having HFPI = 3.69, PEVE content of 0.71 wt%, and $MV = 2.37 \times 10^3$ Pa·s was evaluated by capillary rheometry at 350°C using an Instron® capillary rheometer. Tungsten carbide dies with capillary diameter of 0.0762 cm, capillary length of 2.54 cm, and 90° entrance angle were used. By varying the rate of polymer extrusion through the capillary die, shear rates in the range of from 10.4 s⁻¹ to 3470 s⁻¹ were achieved. The extrudate was observed to be smooth and undistorted over the entire shear rate range studied, exhibiting no sign of melt fracture, even at the highest shear rate attained. In contrast, a TFE/HFP/PPVE terpolymer control resin having HFPI = 3.55, PPVE content of 0.82 wt%, and $MV = 2.44 \times 10^3$ Pa·s exhibited a smooth extrudate at shear rates below 104 s⁻¹, but exhibited sharkskin-like surface melt fracture at shear rates above 104 s⁻¹ that increased in severity with increasing shear rate so that the extrudate became grossly distorted at shear rates above 1000 s⁻¹. This illustrates that the extrusion process of this invention can be operated at high shear rate.

Example 9

[0035] The same TFE/HFP/PEVE terpolymer resin used in Example 8 was evaluated under uniaxial extension at 350°C using a Goettfert Rheotens® Tensile Tester for Polymer Melts. In this test, an evenly extruded melt strand is gripped between two counter-rotating wheels that elongate the strand with constant acceleration until the strand breaks. The velocity of the strand achieved at break is a measure of the extensional properties of the polymer and is an indication of the ability of the polymer to be melt drawn. The polymer was extruded at a shear rate of 9.648 s⁻¹ through a capillary die with capillary diameter 0.2 cm, capillary length 1 cm, and 180° entrance angle to form a melt strand. The strand was extruded vertically downward for a distance of 10.7 cm where it was gripped between two counter rotating wheels that elongated the melt strand with constant acceleration of 0.24 cm/s². The strand elongated smoothly to a final take away velocity of 120 cm/s, the maximum velocity attainable with the available apparatus. In contrast, the TFE/HFP/PPVE terpolymer control resin used in Example 8 elongated smoothly only up to a take away velocity of 51 cm/s, at which point the strand began to neck down and to undergo gross fluctuations in strand thickness. The melt strand eventually broke at a take away velocity of 77 cm/s.

Example 10

[0036] The TFE/HFP/PEVE copolymer resin of Example 8 was used to extrude insulation onto AWG 24 solid copper conductor (20.1 mil = 0.51 mm diameter), using a Nokia-Maillefer 60-mm extrusion wire line in a melt draw extrusion technique. The extruder had length/diameter ratio of 30/1 and was equipped with a conventional mixing screw (See Saxton, U.S. Patent 3,006,029) to provide a uniform melt. Die diameter was 0.32 inch (8.13 mm), guide tip diameter was 0.19 inch (4.83 mm), and land length was 0.75 inch (19 mm). Drawdown ratio was 97. Cone length was 2 inch (51 mm) and the air gap to a water quench was 33 ft (10 m). The temperature profile, other running conditions, and results are shown in Table 2 for extrusions starting at 1500 ft/min (456 m/min) and increasing to 3000 ft/min (914 m/min) in several increments. At higher speed, the process became unstable. The high extrusion speed achieved with very low incidence of spark failures, for thin-walled (0.164 mm) insulation, shows the performance advantage of the TFE/HFP/PEVE copolymer used in this invention. In contrast, similar extrusion of a TFE/HFP/PPVE terpolymer control resin having HFPI = 3.22, PPVE content of 0.93 wt%, and $MV = 2.54 \times 10^3$ Pa·s could be carried out at speeds up to about 1900 ft/min (579 m/min). At higher speed, the process became unstable. Conditions and results are shown in Table 3.

Table 2. Extrusion Summary for Example 10**Temperatures (°F/°C)**

| | | | | | | |
|--------------|-------|---------|-------|------|------|------|
| Rear | ←←←←← | 695/368 | →→→→→ | | | |
| Center rear | ←←←←← | 725/385 | →→→→→ | | | |
| Center | ←←←←← | 735/391 | →→→→→ | | | |
| Center front | ←←←←← | 735/391 | →→→→→ | | | |
| Front | ←←←←← | 740/393 | →→→→→ | | | |
| Clamp | ←←←←← | 740/393 | →→→→→ | | | |
| Adapter | ←←←←← | 740/393 | →→→→→ | | | |
| Crosshead | ←←←←← | 740/393 | →→→→→ | | | |
| Die | ←←←←← | 765/407 | →→→→→ | | | |
| Melt | 757/ | 760/ | 762/ | 767/ | 771/ | 774/ |
| | 403 | 404 | 406 | 408 | 411 | 412 |
| Wire preheat | ←←←←← | 280/138 | →→→→→ | | | |

Running conditions

| | | | | | | |
|--------------------|-----|-----|-----|-----|-----|-----|
| Wire speed (m/min) | 457 | 549 | 610 | 732 | 853 | 914 |
|--------------------|-----|-----|-----|-----|-----|-----|

| | | | | | | |
|-----------------|-----|-----|-----|-----|-----|------|
| Pressure (MPa)* | 7.0 | 8.1 | 8.7 | 9.1 | 9.6 | 11.6 |
|-----------------|-----|-----|-----|-----|-----|------|

Extrudate properties

| | | | | | | |
|--------------------|------|------|------|------|------|------|
| Diameter (mm) | 0.84 | 0.84 | 0.84 | 0.84 | 0.84 | 0.84 |
| Capacitance (pF/m) | 195 | 194 | 194 | 195 | 192 | 187 |
| Length coated (km) | 9.1 | 13.0 | 14.3 | 14.3 | 14.3 | 14.3 |
| Spark failures | 1 | 0 | 2 | 1 | 3 | 4 |

* Pressure at crosshead

Table 3. Extrusion Summary for control B

5

Temperatures (°F/°C)

10

15

20

| | | | | |
|--------------|---------|---------|---------|-----|
| Rear | 688/364 | ←←← | 685/363 | →→→ |
| Center rear | ←←←←← | 720/382 | →→→→→ | |
| Center | ←←←←← | 730/388 | →→→→→ | |
| Center front | 735/391 | ←←← | 730/388 | →→→ |
| Front | 740/393 | ←←← | 730/388 | →→→ |
| Clamp | 745/396 | ←←← | 735/391 | →→→ |
| Adapter | 750/399 | ←←← | 735/391 | →→→ |
| Crosshead | 760/404 | ←←← | 745/396 | →→→ |
| Die | 760/404 | ←←← | 765/407 | →→→ |
| Melt | 757/403 | 764/407 | 765/407 | |
| Wire preheat | ←←←←← | 250/121 | →→→→→ | |

25

Running conditions

30

35

| | | | |
|--------------------|------|------|------|
| Die diameter (mm) | 7.67 | 7.11 | 7.11 |
| Tip diameter (mm) | 4.83 | 4.45 | 4.45 |
| Cone length (mm) | 51 | 38 | 38 |
| Draw down ratio | 99 | 86 | 86 |
| Wire speed (m/min) | 305 | 518 | 579 |
| Pressure (MPa)* | 5.1 | 10.7 | 11.4 |

40

Extrudate properties

| | | | |
|--------------------|------|------|------|
| Diameter (mm) | 0.79 | 0.79 | 0.79 |
| Capacitance (pF/m) | 220 | 179 | 191 |
| Length coated (km) | 9.1 | 18.3 | 18.3 |
| Spark failures | 0 | 4 | 4 |

45

* Pressure at crosshead

Claims

50

1. A process comprising extruding and melt drawing a tetrafluoroethylene copolymer resin to form insulation on an electrical conductor, wherein said copolymer is partially-crystalline copolymer comprising tetrafluoroethylene, hexafluoropropylene in an amount corresponding to an hexafluoropropylene index (HFPI) of from 2.0 to about 5.3 which expresses the ratio of the 10.18 micrometer absorbance to the 4.25 micrometer absorbance as measured with Fourier transform infrared spectroscopy, and from 0.2% to 3% by weight of perfluoro(ethyl vinyl ether).

55

2. The process of Claim 1, wherein the amount of perfluoro(ethyl vinyl ether) in said copolymer is from 0.5% to 2.5% by weight.

3. The process of Claim 2, wherein the amount of said hexafluoropropylene in said copolymer corresponds to HFPI of from 2.8 to 4.1.
4. The process of Claim 1, wherein said copolymer has a melt viscosity of no more than 10×10^3 Pa.s.

5

Patentansprüche

1. Verfahren, umfassend das Extrudieren und Recken aus der Schmelze von Tetrafluorethylen-Copolymerharz, um auf einem elektrischen Leiter eine Isolation zu erzeugen, wobei das Copolymer ein teilkristallines Copolymer ist, aufweisend Tetrafluorethylen, Hexafluorpropylen in einer Menge entsprechend einem Hexafluorpropylen-Index (HFPI) von 2,0 bis etwa 5,3 (womit das Verhältnis des spektralen Absorptionsmaßes von 10,18 Mikrometer zu dem spektralen Absorptionsmaß von 4,25 ausgedrückt wird, gemessen mit Hilfe der FT-Infrarotspektroskopie), sowie von 0,2 bis 3 Gewichtsprozent Perfluor(ethylvinylether),
2. Verfahren nach Anspruch 1, bei welchem die Menge Perfluor(ethylvinylether) in dem Copolymer 0,5 bis 2,5 Gewichtsprozent beträgt.
3. Verfahren nach Anspruch 2, bei welchem die Menge des Hexafluorpropylens in dem Copolymer einem HFPI von 2,8 bis 4,1 entspricht.
4. Verfahren nach Anspruch 1, bei welchem das Copolymer eine Viskosität der Schmelze von nicht mehr als 10×10^3 Pa.s hat.

25

Revendications

1. Procédé comprenant l'extrusion et l'étirage à l'état fondu d'une résine de copolymère de tétrafluoroéthylène pour former une isolation sur un conducteur électrique, dans lequel ledit copolymère est un copolymère partiellement cristallin contenant du tétrafluoroéthylène, de l'hexafluoropropylène en une quantité correspondant à un indice d'hexafluoropropylène(HFPI) de 2 à environ 5,3 qui exprime le rapport de l'absorbance de 10,18 micromètres à l'absorbance de 4,25 micromètres telles que mesurées avec la spectroscopie infra-rouge par transformée de Fourier, et de 0,2 % à 3 % en poids de perfluoro(éthylvinyléther).
2. Procédé selon la revendication 1, dans lequel la quantité de perfluoro(éthylvinyléther) dans ledit copolymère est de 0,5% à 2,5 % en poids.
3. Procédé selon la revendication 2, dans lequel la quantité dudit hexafluoropropylène dans ledit copolymère correspond à un indice HFPI de 2,8 à 4,1.
4. Procédé selon la revendication 1, dans lequel ledit copolymère a une viscosité à l'état fondu de pas plus de 10×10^3 Pa.s.

45

50

55